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(71) Applicant: **Shah, Janak Ramanlal**
Mumbai 400 056 Maharashtra (IN)

(72) Inventors:
• **Tiwari, Manoj Shyamnarayan**
421501 Thane, Maharashtra (IN)

• **Shah, Janak Ramanlal**
400 056 Mumbai, Maharashtra (IN)

(74) Representative: **Engelhard, Markus**
Boehmert & Boehmert
Anwaltpartnerschaft mbB
Pettenkoferstrasse 22
80336 München (DE)

Remarks:

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(54) **A SYSTEM FOR CONTROLLED CONCENTRATION AND SOLIDS RECOVERY**

(57) The present invention relates to a system for one pot solids recovery from solutions, slurries, emulsions, dispersions, gels, semisolids, and their like. Further the system can be used for controlled concentration of solutions, slurries, emulsions, dispersions, gels, semisolids, and their like to enable easy to operate cost effective energy efficient processes. The system is so con-

structed to enhance the contact between the liquid medium and the gaseous medium used in the process for effective heat transfer. The system can be used for controlled concentration and / or recovery of substantially dry solids in applications related to foods, nutraceuticals, natural products, pharmaceuticals, chemicals, etc.

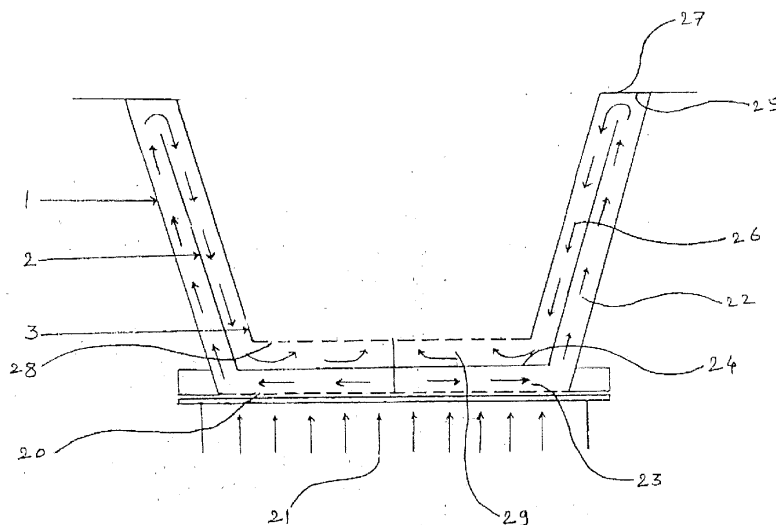


Figure 2

Description

Field of Invention

[0001] The present invention relates to a system for one pot solids recovery from solutions, slurries, gels, semisolids, and their like. Further the system can be used for controlled concentration of solutions, slurries, gels, semisolids, and their like.

Background of the Invention

[0002] Recovery of solids from solutions, slurries, dispersions, emulsions, gels, semisolids, and their like involves the removal of the solvent and / or the carrier medium by diverse processes that involve evaporation, reverse osmosis, ultrafiltration, pervaporation, freeze concentration, clathration etc. Such processes are generally carried out using various types of equipments such as spray driers, drum driers, freeze driers, foam-mat, fluid bed driers, etc.

[0003] Spray driers are routinely used for recovery of solids from solutions / slurries. Similarly fluid bed driers are routinely used to dry wet solids but cannot be used for recovery of solids from solutions, dispersions, slurries, emulsions, gels, semisolids and their like.

[0004] The challenges in technologies related to the treatment of solutions, dispersions, slurries, emulsions, gels, semisolids and their like for the recovery of substantially dry solids are in providing cost effective equipments / systems that enable easy to operate energy efficient processes. The present inventions address this technology gap.

Definition

[0005] The term "substantially dry solids" when used herein shall mean the "loss on drying" to be less than 10% by weight of the solids, preferably less than 5% by weight of the solids, more preferably less than 2% by weight of the solids and most preferably less than 1% by weight of the solids depending on the type and nature of the material and the method used for its determination.

Objects of the Invention

[0006] The main object of the invention is to provide a solid recovery system for the recovery of substantially dry solids from solutions, dispersions, slurries, emulsions, gels, semisolids and their like that enables easy to operate cost effective energy efficient processes.

[0007] Another object of the invention is to provide a system for a single pot process for the concentration of solutions, slurries, dispersions, emulsions, gels, semisolids, and further substantially drying the solids for their recovery.

[0008] Another object of the invention is to provide a system to enhance the contact between the liquid phase

and the gaseous medium used in the process for effective heat transfer for controlled concentration and / or recovery of substantially dry solids from solutions, slurries, dispersions, emulsions, gels, semisolids, and their like.

[0009] Yet another object of the invention is to provide a method of using the said system for controlled concentration and / or recovery of substantially dry solids in applications related to foods, nutraceuticals, natural products, pharmaceuticals, chemicals, etc.

[0010] Thus in accordance with the invention, the system comprises,

a container module, gas pressurizing means, vapor extraction means, and optional filters,

wherein the said container module comprises

first container that is provided with perforated base for passage of pressurized gas,

second container disposed in the said first container so as to form a first continuous annular space on the sides as well as to define space between the base of the said first container and base of the said second container to enable gas flow,

wherein the said first annular space is closed on the top, wherein the base of the said second container has a substantially flat bottom,

third container disposed inside the said second container to define a second continuous annular space on the sides as well as to define the space between the base of the said third container and the base of the said second container

wherein the said second annular space is closed from top, the base of the said third container being provided with perforations or passages;

the base of the said first container is operably connected with the outlet of the pressurized gas source,

the said container module is integrated in a housing with vapor extraction means at the top and an optional filter.

[0011] The liquid medium (solution / dispersion / emulsion / slurry / gel / semisolid and their like) is filled in the said third container, and pressurized gas (for example air) is made to flow from the base of the said first container through the said first annular space into the said second annular space and further from the passages in the base of the said third container to mix with the liquid medium, causing the solvent to evaporate and further dry the formed solids in the same system for recovery.

[0012] In an embodiment of the process, the hot pressurized gas such as air of appropriate temperature and humidity may be used depending on the nature of the solvent / carrier in the solution, slurry, gel, dispersion, emulsion, semisolid etc.

Detailed Description of the Invention

[0013] Features and advantages of the invention will become apparent in the following detailed description and the preferred embodiments with reference to the accompanying drawings.

[0014] The schematic of the system of the invention is

illustrated in **Figure 1**. It comprises of a container module **10**, gas pressurizing means **11**, expansion chamber **12**, filter housing **13**, vapor exhaust (extraction) port **14**.

[0015] The schematic of the container module **10** is illustrated in **Figure 2**. It comprises of first container **1** that is provided with perforated base or perforated gas distribution plate **20** for gas passage. The flow path of the gas such as pressurized air is indicated in the **Figure 2** by arrow **21** for better understanding. The second container **2** is disposed in the said first container **1** so as to define first annular space **22** between first and the second container as well as to define space **23** between the base **20** of the said first container and base **24** of the said second container **2**.

[0016] The said first annular space **22** is closed on the top side **25** as illustrated in the **Figure 2**. The base **24** of the said second container **2** is substantially flat or is provided with a substantially flat plate. The third container **3** is disposed inside the said second container **2** to define second annular cavity / space **26** as well as to define the space **29** between the base **28** of the said third container and the base **24** of the said second container. The said second annular space **26** is closed from top side **27** as illustrated in the **Figure 2**. The base **28** of the said third container **3** is provided with a set of perforations/ passages or is provided with a gas distribution plate with a set of perforations/ passages.

[0017] In one of the embodiments, the said first, second and third containers **1**, **2** and **3** respectively are of frusto-conical geometry.

[0018] The base **20** of the said first container **1** is operably connected with the outlet of the gas pressurizing means such as compressor or blower. The said container module **10** is operably connected to the expansion chamber **12** and further to the filter housing **13** as depicted in **Figure 1**.

[0019] In operation, the solution/ dispersion/ emulsion / slurry / gel / semisolid and their like is filled in the said third container **3**. The pressurized gas flows from the base **20** of the said first container **1** through the said space **23** and further through the first annular space **22** into the said second annular space **26**. It further passes from the passages in the base **28** of the said third container **3** to mix with the solution / dispersion/ emulsion / slurry / gel / semisolid and their like, cause the solvent to evaporate and the dry the resulting solids for final recovery.

[0020] In one of the embodiments, the said third container **3** is provided with a set of inclined passages configured to distribute gas in the bulk of the liquid medium (solution / dispersion/ emulsion / slurry / gel / semisolid and their like) to create turbulence for enhancement of contact between the gas and the liquid medium for improved heat transfer. In one of the variants of this embodiment, the passages are inclined to the horizontal at 15° to 85°, preferably 25° to 75°.

[0021] One of the configurations of the said passages in the said base **28** is depicted in **Figure 3**. As an illustration, only quarter of the gas distribution plate is indi-

cated with the configuration of the passages (individual passage is indicated by numeral **50**). The said passages may be of any shape such as oval, flat oval, rectangular, circular, square, elliptical, or combinations thereof. The ratio of thickness of the said base **28** to the length of the passage is in the range of 0.250 to 0.999.

The ratio of total area of the passages to the area of the base is in the range of 0.01 to 0.50, preferably 0.03 to 0.30, more preferably 0.05 to 0.10.

[0022] In an embodiment of the invention, the said passages are provided with internal serrations to provide swirling motion to the gas passing through it.

[0023] In yet another embodiment the passages provided in the base of the said third container are of diverse cross section such as round, oval, flat oval, rectangular, square etc.

[0024] In yet another embodiment plurality of passages are provided on the sides of the said third container.

[0025] In another embodiment there are more than three containers disposed so as to create more than two annular spaces between them.

[0026] The invention further provides non-limiting examples.

Example 1:

[0027] A solution of 6 kg of sucrose was prepared in 25 kg of water and 5 kg of acetone and filled in third container. The third container was provided with gas distribution plate with passages inclined to the horizontal at 55°. The system was preheated with a stream of hot and dehumidified air. Pressurized hot air was introduced into the system from the bottom of first container and process was run for about 1.5 hours with exhaust kept on to remove the evaporated solvent vapors from the system. The inlet air temperature was about 60°C to 90°C resulting in bed temperature of about 30°C to 55°C and outlet air/vapor temperature of about 30°C to 45°C. The solution gradually got concentrated with the emergence of the solids which got dried as a fluidized bed till the solvent was completely removed and the substantially dry solids were obtained. The solid material was removed and weighed. The yield of the process was 96.3% and the moisture content in the solids was ~0.7%.

Example 2:

[0028] 5 kg of non-pareil seeds was added to 25 kg of purified water. The mixture was stirred to obtain a dispersion which was charged in the third container. The third container was provided with gas distribution plate with passages inclined to the horizontal at 55°. The process described in example 1 was carried out. At the end of the process, solid material was removed and weighed. The yield of the process was ~95% and the moisture content in the solids was less than 1.5%.

Example 3:

[0029] 0.400 kg of starch was added to 1.5 kg of isopropyl alcohol. The mixture was stirred to obtain a dispersion which was filled in third container. The third container was provided with a gas distribution plate with passages inclined to the horizontal at 25°. The system was preheated with a stream of hot and dehumidified air. Pressurized hot air was introduced into the system from the bottom of first container and process was run for about 1 hours with exhaust kept on to remove the evaporated solvent vapors from the system. The inlet air temperature was about 60°C resulting in bed temperature of about 15°C to 55°C and outlet air/vapor temperature of about 20°C to 45°C. The solution gradually got concentrated with the emergence of the solids which got dried as a fluidized bed till the solvent was completely removed and the substantially dry solids were obtained. The solid material was removed and weighed. The yield of the process was ~81% and the moisture content in the solids was about 5.2 %.

Example 4:

[0030] 0.300 kg of povidone K30 was added to 0.13 kg of purified water. The mixture was stirred to obtain a gel. The third container was provided with gas distribution plate with passages inclined to the horizontal at 75°. Purified talc (0.007 kg) was sprinkled (applied) to the inner walls of third container and the upper surface of gas distribution plate. The prepared gel was filled in third container. The system was preheated with a stream of hot remove the evaporated solvent vapors from the system. The inlet air temperature was about 65°C to 85°C resulting in bed temperature of about 35°C to 70°C and outlet air/vapor temperature of about 40°C to 65°C. The solution gradually got concentrated with the emergence of the solids which got dried as a fluidized bed till the solvent was completely removed and the substantially dry solids were obtained. The solid material was removed and weighed. The yield of the process was 90% and the moisture content in the solids was about 2.8%.

Example 5:

[0031] 0.400 kg of lactose was added to 4.89 kg of purified water. The mixture was stirred to obtain a solution which was filled in third container. The third container was provided with gas distribution plate with passages inclined to the horizontal at 35°. The system was preheated with a stream of hot and dehumidified air. Pressurized hot air was introduced into the system from the bottom of first container and process was run for about 3.5 hours with exhaust kept on to remove the evaporated solvent vapors from the system. The inlet air temperature was about 45°C to 90°C resulting in bed temperature of about 25°C to 75°C and outlet air/vapor temperature of about 30°C to 60°C. The solution gradually got concentrated

with the emergence of the solids which got dried as a fluidized bed till the solvent was completely removed and the substantially dry solids were obtained. The solid material was removed and weighed. The yield of the process was 91.75% and the moisture content in the solids was 0.62%.

[0032] The invention described demonstrates the effectiveness of the designed systems that enables a one pot solids recovery from solutions, emulsions, dispersions, slurries, gels, semisolids, and their like. Further the equipment and the process can be used for controlled concentration of solutions, slurries, dispersions, emulsion, semisolids, and gels and materials of their like.

[0033] The invention is now further described by the following embodiments which are given to illustrate, not to limit the present invention.

Embodiments**[0034]**

1. A system for controlled concentration and solids recovery comprising a container module (10), gas pressurizing means (11), vapor extraction means wherein the said container module comprises first container (1) that is provided with perforated base (20) for passage of pressurized gas, second container (2) disposed in the said first container (1) so as to form a first continuous annular space (22) on the sides as well as to define space (23) between the base (20) of the said first container and base (24) of the said second container (2); wherein the said first annular space is closed on the top, wherein the base (24) of the said second container (2) has a substantially flat bottom, third container (3) disposed inside the said second container to define a second continuous annular space (26) on the sides as well as to define the space (29) between the base (28) of the said third container and the base (24) of the said second container wherein the said second annular space (26) is closed from top (27), the base (28) of the said third container being provided with passages (50); the base (20) of the said first container (1) is operably connected with outlet of the pressurized gas source, the said container module (10) is integrated in a housing with vapor extraction means at the top.

2. A system for controlled concentration and solids recovery according to embodiment 1 wherein the said first (1), second (2) and third containers (3) are of frusto-conical geometry.

3. A system for controlled concentration and solids recovery according to any of embodiments 1-2 wherein the said third container (3) is provided with a set of passages (50) configured to distribute gas

in the bulk of the liquid medium to create turbulence for enhancement of contact between the gas and the medium for improved heat transfer.

4. A system for controlled concentration and solids recovery according to any of embodiments 1-3 wherein the passages (50) are inclined to the horizontal at 15° to 85°.

5. A system for controlled concentration and solids recovery according to any of embodiments 1-4 wherein the gas pressurizing means is compressor or blower.

6. A system for controlled concentration and solids recovery according to any of embodiments 1-5 wherein the said container module (10) is operably connected to the expansion chamber (12) and further to the filter housing (13).

7. A system for controlled concentration and solids recovery according to any of embodiments 1-6 wherein the passages are of oval, flat oval, rectangular, circular, square, elliptical or a combination thereof shape.

8. A system for controlled concentration and solids recovery according to any of embodiments 1-7 wherein the ratio of thickness of the said base (28) to the length of the passage is in the range of 0.250 to 0.999.

9. A system for controlled concentration and solids recovery according to any of embodiments 1-8 wherein the ratio of total area of the passages to the area of the base is in the range of 0.01 to 0.50.

10. A system for controlled concentration and solids recovery according to any of embodiments 1-9 wherein the said passages are provided with internal serrations to provide swirling motion to the gas passing through it.

11. A system for controlled concentration and solids recovery according to any of embodiments 1-10 wherein the passages provided in the base of the said third container (3) are of diverse cross section such as round, oval, flat oval, rectangular, square or a combination thereof.

12. A system for controlled concentration and solids recovery according to any of embodiments 1-11 wherein plurality of passages are provided on the sides of the said third container.

13. A system for controlled concentration and solids recovery according to any of embodiments 1-12 wherein more than three containers are disposed so

as to create more than two annular spaces between them.

14. A system for controlled concentration and solids recovery according to any of embodiments 1-13 wherein the gas is air.

Claims

1. A method for controlled concentration and/or recovery of substantially dry solids, wherein the method comprises:

a) providing a system for the recovery of substantially dry solids, said system comprising a container module, gas pressurizing means, vapor extraction means, and optional filters, wherein said container module comprises a first container that is provided with perforated base for passage of pressurized gas, a second container disposed in said first container so as to form a first continuous annular space on the sides as well as to define space between the base of said first container and the base of said second container to enable gas flow, wherein said first annular space is closed on the top, wherein the base of said second container has a substantially flat bottom, a third container disposed inside said second container to define a second continuous annular space on the sides as well as to define the space between the base of said third container and the base of said second container, wherein said second annular space is closed from top, the base of said third container being provided with perforations or passages; the base of said first container is operably connected with the outlet of the pressurized gas source, said container module is integrated in a housing with vapor extraction means at the top and an optional filter;

b) filling a liquid medium selected from solution, dispersion, emulsion, slurry, gel or semisolid into said third container;

c) making a pressurized gas to flow from the base of said first container through said first continuous annular space into said second continuous annular space and further from the passages in the base of said third container to mix with said liquid medium;

d) causing the solvent to evaporate to concentrate the liquid medium and drying the solids in said system for recovery.

2. The method according to claim 1, wherein the liquid medium is a solution.

3. The method according to claim 1, wherein the liquid medium is a dispersion.
4. The method according to claim 1, wherein the liquid medium is a gel. 5
5. The method according to any of claims 1 - 4, wherein, by a set of inclined passages in said third container, said pressurized gas is distributed in the bulk of the liquid medium to create turbulence for enhancement of contact between the gas and the liquid medium for improved heat transfer. 10
6. The method according to any of claims 1 - 5, wherein said gas pressurizing means is a compressor or blower. 15
7. The method according to any of the foregoing claims, wherein said pressurized gas is air. 20

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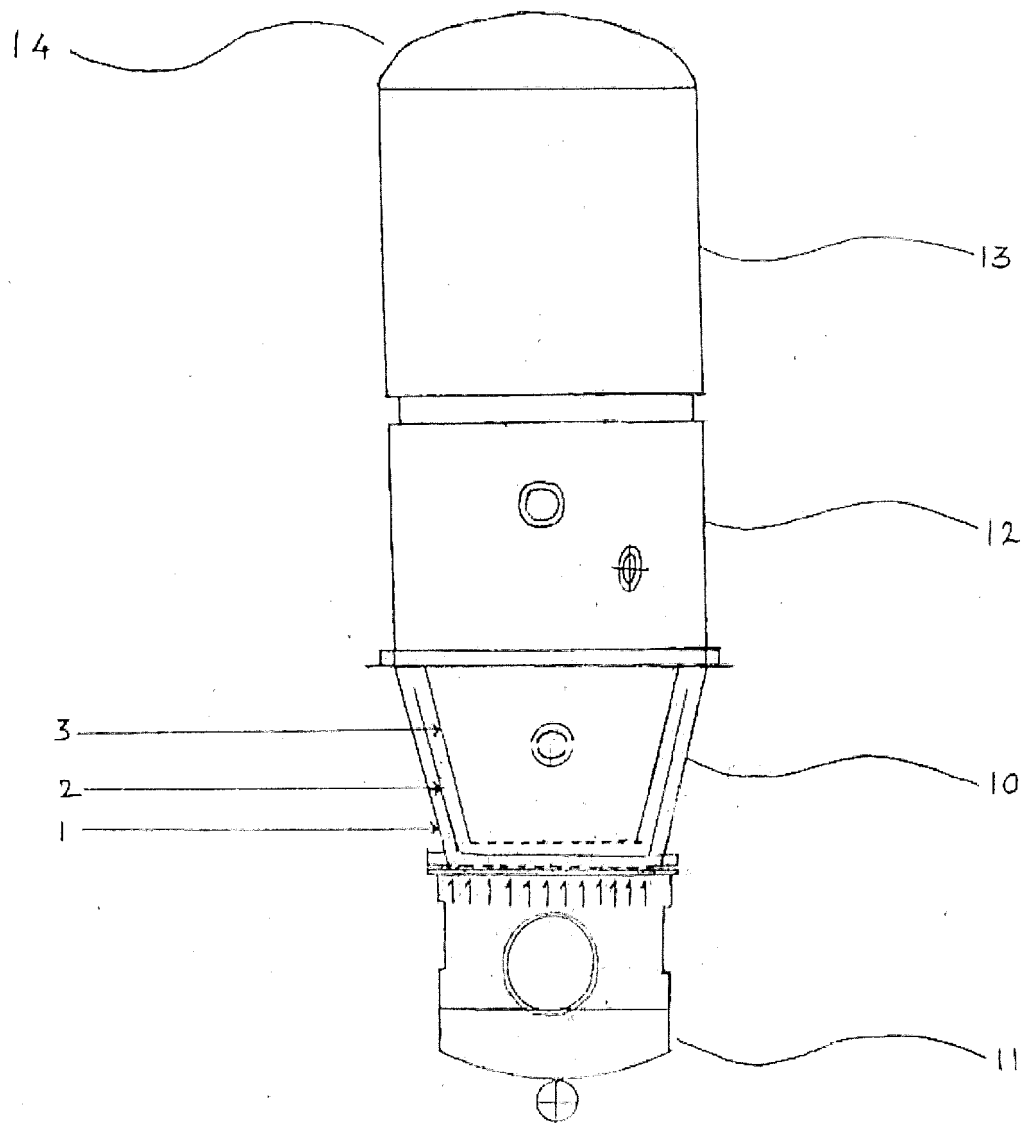


Figure 1

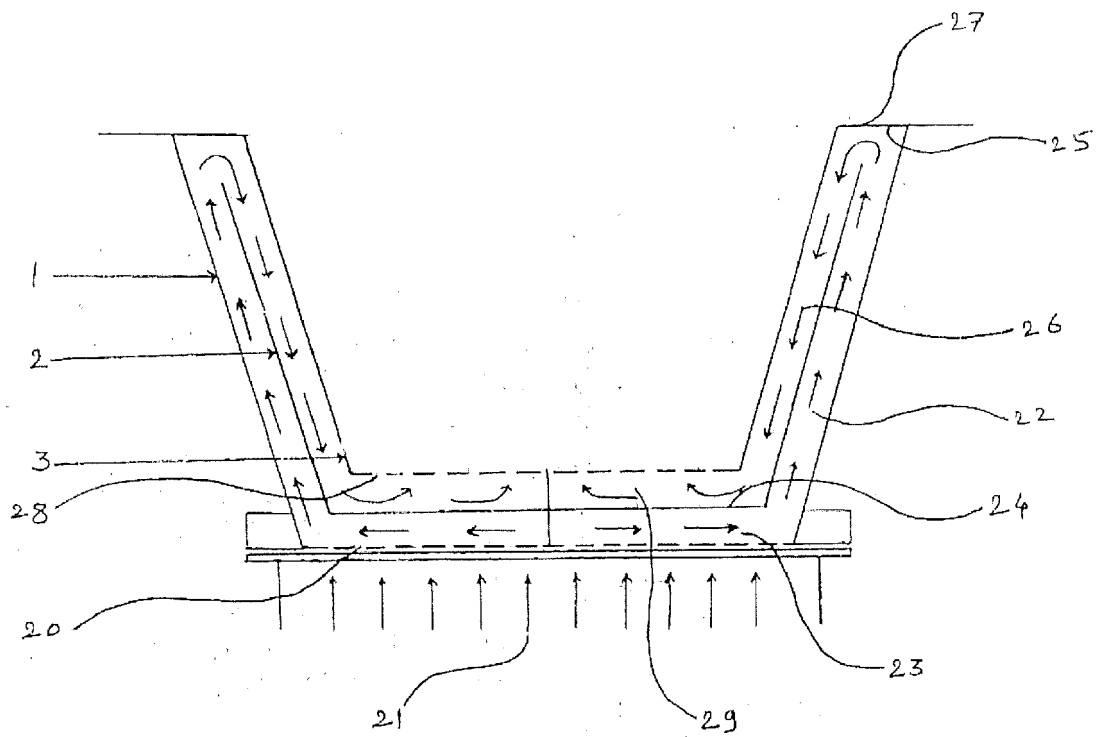


Figure 2

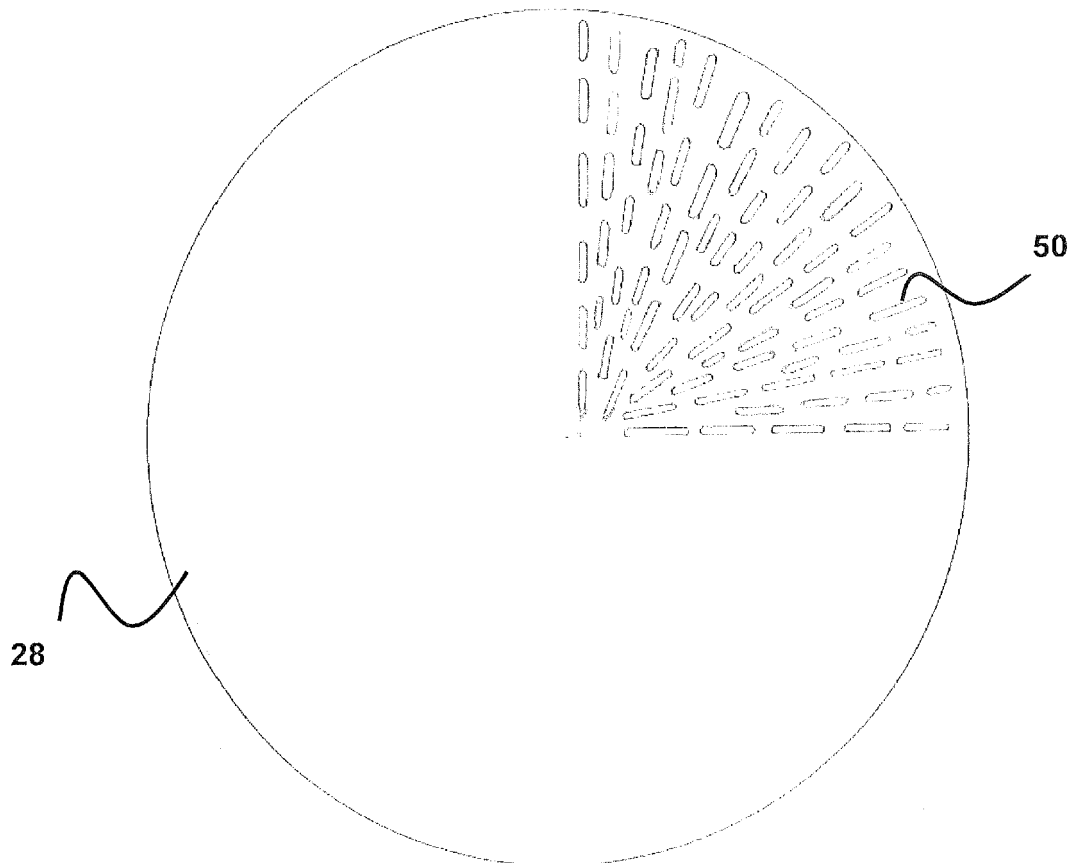


Figure 3



EUROPEAN SEARCH REPORT

Application Number
EP 17 17 7667

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A	JP H05 220314 A (MAEKAWA SEISAKUSHO KK) 31 August 1993 (1993-08-31) * abstract *	1-7	INV. F26B17/10 F26B21/00 F26B25/10 F26B25/18 B01D1/18
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			TECHNICAL FIELDS SEARCHED (IPC)
			F26B
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
The Hague		13 September 2017	Haegeman, Marc
CATEGORY OF CITED DOCUMENTS			
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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 17 17 7667

5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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13-09-2017

10	Patent document cited in search report	Publication date	Patent family member(s)	Publication date
	JP H05220314 A	31-08-1993	NONE	

15	JP 2006266552 A	05-10-2006	NONE	

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